



A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

Working with Hazardous Chemicals

The procedures in *Organic Syntheses* are intended for use only by persons with proper training in experimental organic chemistry. All hazardous materials should be handled using the standard procedures for work with chemicals described in references such as "Prudent Practices in the Laboratory" (The National Academies Press, Washington, D.C., 2011; the full text can be accessed free of charge at http://www.nap.edu/catalog.php?record_id=12654). All chemical waste should be disposed of in accordance with local regulations. For general guidelines for the management of chemical waste, see Chapter 8 of Prudent Practices.

In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior to performing a reaction, a thorough risk assessment should be carried out that includes a review of the potential hazards associated with each chemical and experimental operation on the scale that is planned for the procedure. Guidelines for carrying out a risk assessment and for analyzing the hazards associated with chemicals can be found in Chapter 4 of Prudent Practices.

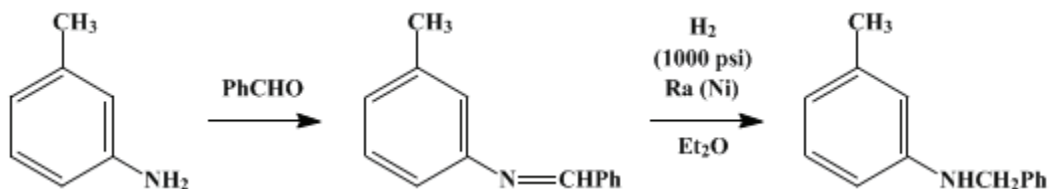
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

Organic Syntheses, Coll. Vol. 3, p.827 (1955); Vol. 21, p.108 (1941).

***m*-TOLYL BENZYLAMINE**

[*m*-Toluidine, N-benzyl]



Submitted by C. F. H. Allen and James VanAllan.

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1. Procedure

One hundred and six grams (1 mole) of benzaldehyde (Note 1) and 107 g. (1 mole) of *m*-toluidine are mixed in a suitable flask; the temperature rises to about 60° (Note 2) and (Note 3). The mixture is cooled below 35° in cold water, 200 ml. of ether is added, and the solution is placed in the steel reaction vessel of a high-pressure hydrogenation apparatus. Eight to ten grams of Raney nickel catalyst (p. 181) is added, the bomb is closed, and hydrogen is admitted up to 1000 lb. pressure (Note 4). The bomb is shaken continuously at room temperature for 15 minutes (Note 5). The contents are removed, and the bomb is washed out with two 200-ml. portions of ether. After the catalyst has been separated by filtration (Note 6), the ether is removed by distillation and the product is distilled from a modified Claisen flask (Note 7). After a small fore-run the *N*-benzyl-*m*-toluidine boils at 153–157° /4 mm.; 315–317° /760 mm. The yield is 175–185 g. (89–94%) (Note 8) and (Note 9).

2. Notes

1. Technical grades of benzaldehyde (b.p. 57–59° /8 mm.) and *m*-toluidine (b.p. 76–77° /7 mm.) are satisfactory.
2. It is unnecessary to isolate the Schiff base.
3. The ether used as solvent may be replaced by an equal amount of 95% ethanol. If ethanol is used, cooling is unnecessary.
4. The initial pressure of hydrogen used is determined by the apparatus available. The drop in pressure depends upon the size of the bomb.
5. With a less active Raney nickel catalyst it may be necessary to carry out the reduction at a somewhat higher temperature. If necessary, the reaction mixture may be heated slowly to 60° and the reduction carried out at this temperature. The yield is then somewhat lower (82–84%).
6. The catalyst is filtered through paper on a Büchner funnel. Owing to its activity, suction should be discontinued as soon as all the liquid has passed through. If this precaution is not observed, a fire may result.
7. In some instances (*o*-tolylbenzylamine), a part of the product may crystallize and can be filtered.
8. The hydrochloride, m.p. 198–199°, may serve for characterization.
9. According to the submitters, other aromatic aldehydes and amines may be used in a similar manner with essentially the same yield of product. The exact procedure for isolation of the amine will depend upon its physical properties. Benzaldehyde and *o*-toluidine yield *o*-tolylbenzylamine, m.p. 56–57° (hydrochloride, m.p. 165–166°), while *p*-tolylbenzylamine, obtained from *p*-toluidine, has a b.p. 162–163°/5 mm. (hydrochloride, m.p. 181–182°).

3. Discussion

N-Benzyl-*m*-toluidine has been prepared by the electrolytic reduction of benzal-*m*-toluidine,¹ and by hydrolysis of *N*-benzyl-*N*-formyl-*m*-toluidine, which was prepared in turn by the reaction of sodium *N*-formyl-*m*-toluidine with benzyl chloride.²

References and Notes

1. Law, *J. Chem. Soc.*, **1912**, 154.
 2. Grunfeld, *Bull. soc. chim. France*, (5) **4**, 654 (1937).
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Appendix Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

sodium N-formyl-m-toluidine

[ethanol \(64-17-5\)](#)

[ether \(60-29-7\)](#)

[hydrogen \(1333-74-0\)](#)

[benzaldehyde \(100-52-7\)](#)

[Raney nickel \(7440-02-0\)](#)

[benzyl chloride \(100-44-7\)](#)

[p-toluidine \(106-49-0\)](#)

[o-toluidine \(95-53-4\)](#)

[m-toluidine \(108-44-1\)](#)

[m-Tolylbenzylamine,
N-benzyl-m-toluidine,
m-Toluidine, N-benzyl \(5405-17-4\)](#)

[o-tolylbenzylamine \(5405-13-0\)](#)

[p-tolylbenzylamine](#)

[benzal-m-toluidine](#)

[N-benzyl-N-formyl-m-toluidine](#)

[N-benzyl-m-toluidine hydrochloride](#)

[o-tolylbenzylamine hydrochloride](#)

[p-tolylbenzylamine hydrochloride](#)